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RESEARCH

Effect of Aluminium Oxide (Al_2O_3) Nanoparticles Addition into Lubricating Oil on Tribological Performance

S. Ghalme[a,*], P. Koinkar[b], Y. Bhalerao[c]

[a] *Mechanical Engineering, Sandip Institute of Technology & Research Centre, Nashik (MS), India,*
[b] *Optical Nanomaterials Laboratory, Tokushima University, Japan,*
[c] *Mechanical Engineering & Design, School of Engineering, University of East Anglia, United Kingdom.*

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* Corresponding author:

Sachin G. Ghalme
E-mail: sachinghalme@hotmail.com

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A B S T R A C T

Minimizing friction and wear in between mating parts is the main concern in the field of tribological studies. Lubricants with improved tribological properties are continuously explored for minimizing friction and wear. In the present work, an attempt has been made to evaluate the effect of the addition of aluminium oxide (Al_2O_3) nanoparticles as lubricant additives on the tribological performance of base lubricant oil (SAE10W40) using four-ball tester. Weight in the percentage of Al_2O_3 nanoparticles was added in base oil and evaluated the effect of additives on wear preventive characteristic and coefficient of friction of base lubricant oil.
Integrated Taguchi-Grey relational approach is implemented to obtain the optimum combination of load and % wt. of Al_2O_3 nanoparticle addition for improving the tribological performance of base lubricant oil. With a load of 250 N and 0.5 wt% of Al_2O_3 nanoparticles shows an optimum combination for the improved tribological performance of base oil. The wear scar diameter and coefficient of friction found to be reduced by 20.75 % and 22.67 % respectively with the addition of 0.5 wt% of Al_2O_3 nanoparticles in base lubricant oil. The lubrication performance seems to be improved because of mending effect and ball bearing effect of Al_2O_3 nanoparticles forming a self-protective film on the friction surface.

1. INTRODUCTION

The study of interacting surfaces is essential to understand the science behind friction and wear of surfaces. In general, tribology is a science of understanding, controlling and managing friction and wear through the mean of lubrication. Friction and wear between interacting surfaces lead to:

1. Failure of components: like gears, bearings, etc., are important for the smooth operation of mechanical systems.
2. Energy loss: high value of friction leads to major loss of energy in various systems. Various studies revealed that about 1/3rd energy generated goes to wastage because

of frictional losses in an engine other movable parts like transmission, brakes and tires [1].

The solution to both issues of failure of components and energy loss is to apply appropriate lubricant in between the contacting surfaces having desirable properties satisfying the operating conditions. Lubricants are used to minimize friction and wear to maintain the required surface quality. Lubricants try to maintain fluid film between contacting surfaces and help minimize friction along with to carry out generated heat and wear particles away. Emerging technologies and applications requires intense and widespread properties from lubricants. To satisfy the required operating conditions, there is a need for enhancement of lubricant properties. Enhancing the lubricant properties through various ways is a key and important area of research in the field of tribology. Several studies are focused on discovering various additives for lubricating oil. A few weight percentages of additives are added to the base oil to enhance the properties, they are essential for improving the tribological performance of lubricants [2]. Additive improves the lubricating properties of base oil by enhancing desirable properties already present in a base oil or adding certain new properties required for base oil.

Nanotechnology was first introduced in 1959 by Nobel laureate Richard P. Feynman during his lecture 'There is plenty of room at the bottom' [3]. Application of nanotechnology encompasses various fields of science, engineering, biomedical and materials science etc. Numbers of researcher have already presented the applicability of nanoparticles addition to lubricant as an efficient mean for improving the tribological performance of lubricant [4-6]. Various studies have presented nanoparticles as a promising additive for improving tribological properties of lubricating oil [7-9]. Nano-additives offer improvement in tribological properties through the four ways [10-13]:

1. Rolling of nano-spheres.
2. Tribochemical reaction forming tribofilm.
3. Minimal size creating mending effect, and
4. Polishing.

In some cases, nanoparticle along with metal debris gets accumulated in valleys over the surface and thereby smoothing the surface with minimizing the coefficient of friction. While hard nanoparticles may plough the surface thereby a higher value of the coefficient of friction and wear [14].

Aluminium oxide (Al_2O_3) or alumina nanoparticles are corundum like structure and having oxygen atom adopting hexagonal close packing in alumina ions [15].

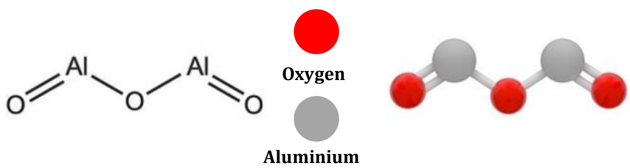

Fig. 1. Aluminium Oxide Structure [16].

Alumina nanoparticles have wide application as an abrasive material, as biomaterials and as a reinforcement in the metal-matrix composite. Al_2O_3 nanoparticles have the capability for the formation of protective film with mending effect and thereby reducing friction and wear [17]. T. Luo *et al.* [18] prepared Al_2O_3 nanoparticles with the hydrothermal method and modified using a silane agent for proper dispersion into a base oil. They evaluated the tribological performance of Al_2O_3 nanoparticles as lubricant additives using four-ball and thrust ring friction test. The experimental result presented 0.1 wt% as optimal addition in base oil reducing friction coefficient by 17.61 % and 23.92 % in four-ball and thrust ring friction test respectively along with 41.75 % reduction in wear scar diameter for four-ball test. A. Thakre and A. Thakur [19] evaluated the effect of Al_2O_3 nanoparticles size (40-80 nm) and its concentration (0-1 %) suspended in SAE20W40 base oil over the coefficient of friction using pin-on-disc tribo-tester. They reported the optimal combination of 0.8 % concentration of Al_2O_3 nanoparticles of 60 nm size in base oil at 1200 rpm and 160 N load to improve the tribological performance of base oil significantly. K. Suthar *et al.* [20] tested tribological performance of jojoba oil with 0.05 to 0.2 % concentrations of Al_2O_3 nanoparticles as additives with pin-on-disc tribotester; they reported 0.1 % concentration is optimal for tribological properties of jojoba oil with reducing the coefficient of friction in mating pair. In another study considering an automotive application to minimize friction between piston ring cylinder assembly M.K.A. Ali *et al.* [20] evaluated the tribological performance of Al_2O_3/TiO_2 nanoparticles suspended in engine oil (0.05, 0.1, 0.25, and 0.5 wt%) using bench tribometer designed to mimic the sliding reciprocating motion of the piston ring-cylinder interface. They reported improvement in tribological properties of engine oil with 0.25 wt% of Al_2O_3/TiO_2 nanoparticles additive in terms of decreasing wear rate, coefficient of friction and frictional power loss. Lubricants have an indirect effect over the fuel economy through the application of engine oil in terms of effect over frictional power loss. M.K.A. Ali *et al.* [22] studied the effect of Al_2O_3/TiO_2 nano additives in engine oil lubricant in view of fuel economy of a gasoline engine. They evaluated engine performance using AVL dynamometer under different operating conditions. The study revealed the improvement in engine efficiency by 1.7-2.5 % with the use of hybrid nano additives in engine oil compared to engine oil without nano additives. The thermal degradation of the base at elevated temperature is major issue for the use of lube oil at higher temperature conditions. M.K.A. Ali and H. Xianjun [23] evaluated the thermal stability of lube oil with Al_2O_3/TiO_2 nano additives using TGA thermal analyzer. The study reported the delayed oxidation onset temperature and burnout temperature of lube oil along with enhancement in heat transfer rate and improving brake thermal efficiency of engine also with the use of hybrid nano additives in lube oil. In another interesting study related to Al_2O_3 nanoparticles as nano additives for MQL is presented by M. Jamil *et al.* [24] and H. Hegab *et al.* [25]. In both studies the lubricant was added with alumina (Al_2O_3) with multi-walled carbon nanotubes (MWCNTs) nano additives in vegetable oil and implemented as MQL for machining of Ti–6Al–4V and Inconel 718 respectively. The results revealed the significant improvement in the properties of the machined component along with tool performance and chip morphology.

The limited amount of work has been reported over the tribological performance of Al_2O_3 nanoparticles as lubricating additives. Most of the researchers have evaluated the effect of size of nanoparticles and the concentration of nanoparticles in a base oil. But, load in contacting pair also has a significant effect on the wear performance of pair. In this study, an attempt has been made to obtain the optimum proportion of Al_2O_3 nanoparticles wt% addition with respect to load for improving wear preventive and friction reduction characteristic through Taguchi-grey relational analysis.

2. EXPERIMENTAL DETAILS

2.1 Materials Used

The present study is aimed towards the evaluation of improvements in tribological properties of base lubricant oil with the addition of Al_2O_3 nanoparticles.

Base oil: Commercially available 10W40 grade base oil purchased from the market for the preparation of nano-lubricant with the addition of Al_2O_3 nanoparticles. It is commonly used lubricant for gearboxes. Table 1 shows the properties of SAE10W40 oil.

Table 1. Properties of SAE10W40 Oil.

Density at 15.6 °C	872 Kg/m^3
Kinematic viscosity at 40 °C	108.5 cSt
Kinematic viscosity at 100 °C	15.4 cSt
CCS viscosity at -25 °C	6270 cP
Flash point	220 ^{0}C
Pour Point	-33 ^{0}C

Additives: Alpha Al_2O_3 nanoparticles with 99.9 % purity and 50 nm size were purchased from the market as an additive for lubricant oil.

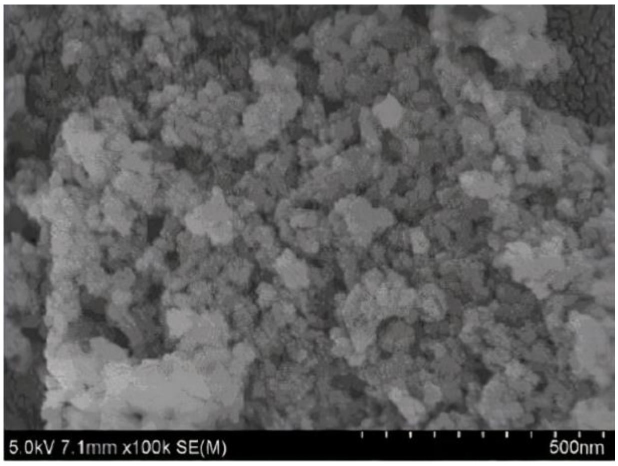

Fig. 2. SEM Image of Al_2O_3 Nanoparticles.

Preparation of nano-lubricant: Aggregation of nanoparticles in the base is a major issue with the preparation of nano-lubricants and it limits

its lubricating ability [26]. The proper dispersion of nanoparticles in the base oil is important. Various coupling/dispersant agents including Aliquat 336 [27], Estisol 242, oleic acid [28], and sorbitol monostearate [29] are used for improving dispersion of nanoparticles in a base oil, theses coupling agents' changes surface properties of nanoparticles from hydrophilic to lipophilic and improves lubrication performance [30]. Three different proportion of Al_2O_3 nanoparticles along with oleic acid as a dispersant agent mixed with a base oil and sonicated using bath ultrasonic for 2hrs. Stability of samples was observed for 24hrs and then used for testing.

2.2 Method: Design of Experiments

Design of experiment-Taguchi method of experimental design helps to design efficient, systematic and optimized experimental plan. The optimization of multiple outputs characteristic is easily possible with grey relational analysis. Grey relational analysis is used to convert multiple outputs into one numerical score and then decide the optimal combination of the parameter using this score [31]. In this study, we implemented Taguchi orthogonal array to plan experiments and grey relational analysis implemented to analyze the experimental results for obtaining an optimal combination of carbon nanotube additive and load for minimizing wear scar diameter and coefficient of friction.

Load and wt% of Al_2O_3 nanoparticles are two factors selected at three levels as shown in Table 2.

Table 2. Designed experimental factors and levels.

Factor	Level 1	Level 2	Level 3
Load (N)	250	500	750
wt% of Al_2O_3	0	0.5	0.75

Based on a number of levels and factors, L9 orthogonal array selected with 9 numbers of experiments.

2.3 Tribological Testing

Four-ball tribotester is a more preferable tester for evaluating tribological properties of any lubricant. Tribological tests were conducted using Ducom made TR 30L four-ball tester to evaluate the wear preventive (WP) and friction characteristic of nano-lubricant. Four ball test machine was configured according to the Institute of Petroleum (UK) IP 300 rolling test procedure. Steel balls of 12.7mm Diameter, made from AISI E52100, hardened to 64-66 HRC steel were used during testing. TR 30L machine comes with WinDucom software for data acquisition and display of results. Figure 3 shows the loading arrangement of four-ball tester. Coefficient of friction was recorded with a data acquisition system and wear scar diameter was measured with image acquisition attachment, and then average wear scar diameter is obtained.

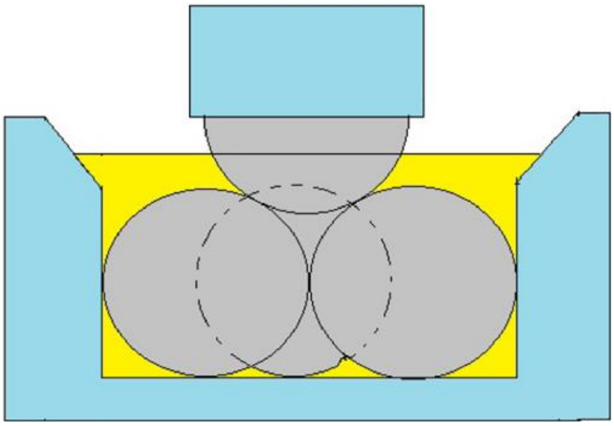

Fig. 3. Schematic illustration of four-ball tester.

Total 9 tests were conducted for three formulation and three loading conditions according to ASTM D4172 and ASTM D5183. Tests were conducted at rolling speed of 1200 rpm, oil bath temperature 75 ^{0}C, and time of 60 min. Wear scar diameter on stationary balls were measured to characterize the worn surface. An optical microscope with 0.01 mm accuracy was used for this purpose. Figure 4 shows sample images of wear scar diameter obtained during testing.

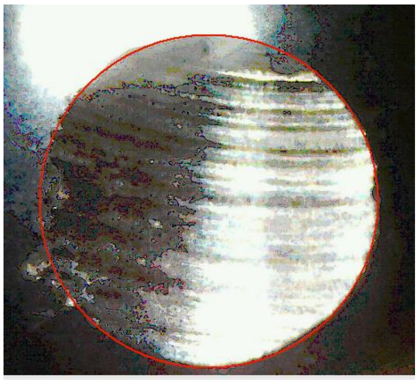

Fig. 4. Sample Wear Scar Diameter Images on Ball Surface.

3. RESULTS AND DISCUSSION

Tests were conducted as per L9 orthogonal array repeating each test two times and during each test average value of wear scar diameter and coefficient of friction was measured. Table 3 shows the experimental plan along with the average value result of each experiment.

Table 3. Experimental plan and results.

Sr. No.	Load (N)	Additives (wt% Al_2O_3 Nanoparticles)	Avg. Wear Scar Dia. (mm)	CoF
1	250	0	0.133	0.085
2	250	0.5	0.063	0.062
3	250	0.75	0.303	0.065
4	500	0	0.401	0.083
5	500	0.5	0.375	0.064
6	500	0.75	0.557	0.07
7	750	0	0.58	0.083
8	750	0.5	0.563	0.068
9	750	0.75	0.72	0.063

It is clear from the experimental results (Table 3) that 0.5 wt% of Al_2O_3 nanoparticle addition in base oil has significant effect on coefficient of friction and wear scar diameter in all loading conditions. In order to consider the integrating effect of load and Al_2O_3 nanoparticle addition on tribological performance of base oil we implemented grey relational analysis. Grey relational analysis helps to convert multiple response problems into a single response problem using Grey relational grade. Numbers of researchers have successfully used integrated Taguchi-Grey approach in the field of tribological study [31-33]. Grey relational analysis applied to convert two output characteristic into a single numerical score to obtain the optimal combination of load and %wt. of Al_2O_3 nano additive. The step-wise Grey relational analysis as follow:

3.1 Grey Relational Analysis

Normalization of Data:
The collected raw experimental data is normalized into 0 or 1, with two criteria wise lower is better (LB) and higher is better (HB). A LB criterion is used to normalize data when the objective function is to minimize. Equation 1 is used for LB criteria. A HB criterion is used to normalize data when the objective function is to maximize. Equation 2 is used for HB criteria.

$$x_i(k) = \frac{\max y_i(k) - y_i(k)}{\max y_i(k) - \min y_i(k)} \tag{1}$$

$$x_i(k) = \frac{y_i(k) - \min y_i(k)}{\max y_i(k) - \min y_i(k)} \tag{2}$$

where $x_i(k)$ is the value after the grey relational generation, $\min y_i(k)$ is the smallest value of $y_i(k)$ for the k^{th} response, and $\max y_i(k)$ is the largest value of $y_i(k)$ for the k^{th} response. $i = 1, 2, 3\ldots$ the number of experiments and $k = 1, 2, 3\ldots$ the number of responses.

In this study, we were expecting to minimize both wear scar diameter as improved wear preventive characteristic and coefficient of friction. So, equation 1 is used to normalize experimental data.

Calculation of Grey Relational Coefficient (GRC):
GRC is calculated to determine the relation between ideal and actual normalized experimental data. GRC (ξ) is calculated using equation 3. A relation is established between actual values and normalized values of wear scar diameter and coefficient friction using equation 3.

$$\xi = \frac{\Delta_{\min} + \psi \Delta_{\max}}{\Delta_{oi}(k) + \psi \Delta_{\max}} \tag{3}$$

where,

$$\Delta_{oi}(k) = \left\| x_o(k) - x_i(k) \right\|$$

The difference of the absolute value of $x_0(k)$ and $x_i(k)$; ψ is the distinguishing coefficient; $0 < \psi < 1$, Δ_{min} is the smallest value of $\Delta_{oi}(k)$ and Δ_{max} is the largest value of $\Delta_{0i}(k)$.

Calculation of Grey Relational Grade (GRG):
The analysis of multiple outputs characteristic is based on grey relational grade. This will convert multiple responses in single numerical value. The GRG (γ) is an average sum of GRC and calculated using equation 4. Its value lies between 0 and 1.

$$\gamma_i = \frac{1}{n} \sum_{k=1}^{n} \xi_i(k) \tag{4}$$

where n are a number of process responses. In this work, the wear scar diameter and coefficient of friction are two responses.

The experimental data is processed using above equations 1 to 4 and converted in grey relation grade (GRG) as presented in Table 4. GRG is calculated considering equal weightage to both responses.

Table 4. Grey relational grade analysis.

Sr. No.	Load (N)	Additives (wt% Al_2O_3 Nanoparticles)	Avg. Wear Scar Dia. (mm)	CoF	Wt. GRG	Rank
1	250	0	0.133	0.085	0.289	5
2	250	0.5	0.063	0.062	0.5	1
3	250	0.75	0.303	0.065	0.343	2
4	500	0	0.401	0.083	0.212	8
5	500	0.5	0.375	0.064	0.341	3
6	500	0.75	0.557	0.07	0.247	7
7	750	0	0.58	0.083	0.186	9
8	750	0.5	0.563	0.068	0.264	6
9	750	0.75	0.72	0.063	0.314	4

From Table 4, it is clear that highest value of weighted grey relational grade with rank 1 occurs at the combination of 0.5 wt% of Al_2O_3 nanoparticles and 250 N load, presenting it as an optimal combination for minimizing wear scar diameter and coefficient of friction.

3.2 Effect of Individual Input Factor

The grey relational analysis presents the optimal combination of input factors for minimizing or maximizing the number of inputs at a time. But, when we will look into the individual effect of load and wt% of Al_2O_3 nanoparticles, it presents significantly different values for load and nano particle addition. This can be visible through the main effect plot as shown in Figs. 5 and 6.

From Figs. 5 and 6 it is clear that at a load of 250 N, we observe the minimum value of wear scar diameter and minimum value of the coefficient of friction. It is evident to the laws of friction and wear that wear scar diameter increases with increase in load. But, in case of the coefficient of friction initially increases with a load of 250 N to 500 N and decreases for 750 N.

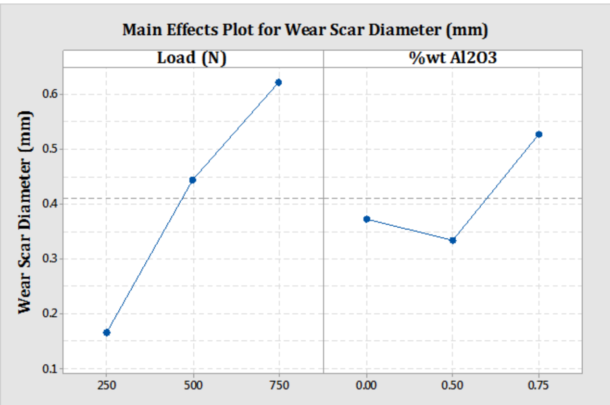

Fig. 5. Main Effect Plot for Wear Scar Diameter.

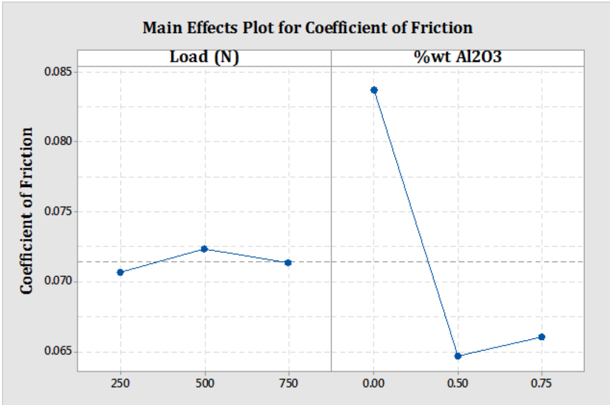

Fig. 6. Main Effect Plot for Coefficient of Friction.

With an increase in load, the deformation in asperities and surface peaks leads to smoothening of surface and minimizing the coefficient of friction. While 0.5 wt% of Al_2O_3 nanoparticles in lubricant offers the minimum value of wear scar diameter and coefficient of friction. With 0.5 wt% of nanoparticles, they act as a spacer between asperities and converts sliding effect into rolling effect due to its ball-bearing effect and mending effect leading to minimizing the coefficient of friction. But with an increase in nanoparticles concentration, the large amount of nanoparticles minimizes ball-bearing effect due to obstruction and locking movement of nanoparticles leading to rise in wear rate and coefficient of friction.

3.3 Effect on Individual Response Parameter

The interacting effect of load and Al_2O_3 nanoparticles on individual wear scar diameter and coefficient of friction is interesting to observe.

Effect on Wear Scar Diameter:
Figure 7, presents the interacting effect of load and Al_2O_3 nanoparticles on wear scar diameter.

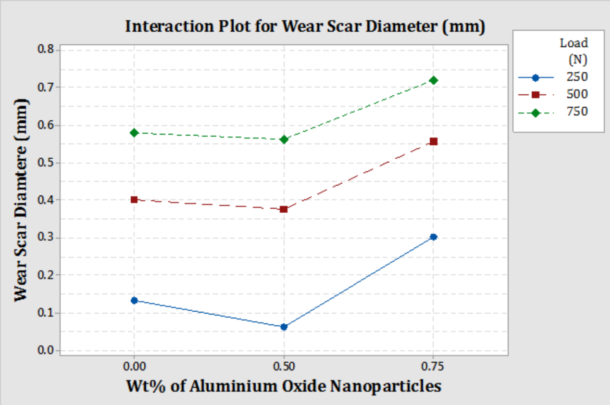

Fig. 7. Interaction Plot for Wear Scar Diameter.

It is clear from the figure that 250N load and 0.5 wt% of Al_2O_3 nanoparticles in lubricant offers the minimum value of wear scar diameter. For all loading conditions, 0.5 wt% of Al_2O_3 nanoparticles offer minimum value of wear scar diameter, suggesting as an improvement in wear preventive characteristic of lubricant. With 0.5 wt% of Al_2O_3 nanoparticles addition in the lubricant, we can observe average reduction in wear scar diameter by 17.42 %.

Effect on Coefficient of Friction:
Figure 8, presents the interacting effect of load and Al_2O_3 nanoparticles on the coefficient of friction.

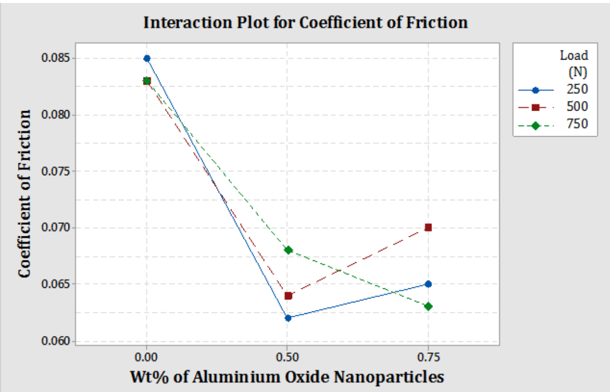

Fig. 8. Interaction Plot for Coefficient of Friction.

It is clear from the figure that 250N load and 0.5 wt% of Al_2O_3 nanoparticles in lubricant offers the minimum value of the coefficient of friction. For all loading conditions, 0.5 wt% of Al_2O_3 nanoparticles offer minimum value of the coefficient of friction except 750N, suggesting as an improvement in friction reduction characteristic of lubricant. With 0.5 wt% of Al_2O_3 nanoparticles addition in lubricant, we can observe average reduction in coefficient of friction by 22.67 %. Also, lines of interaction plot are crossing each other suggesting strong interaction between load and wt% addition of Al_2O_3 nanoparticles addition.

3.4 Integrated Effect on Response Parameter

Integrated effect on response parameters, wear scar diameter and coefficient of friction is evaluated through grey relational analysis. Figure 9 presents the interaction plot for a grey relational grade (GRG).

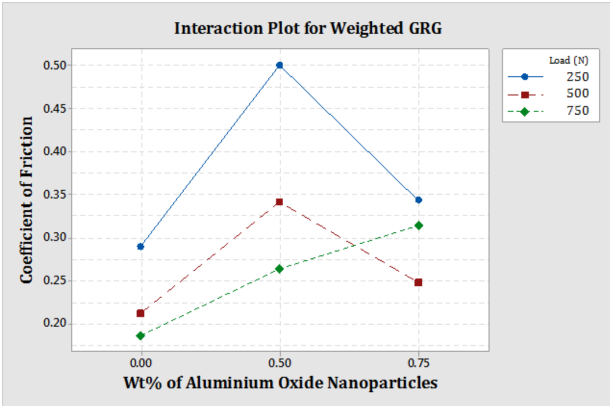

Fig. 9. Interaction Plot for Weighted GRG

Grey relational grade helps to convert multiple responses into a single numerical value. In this study, two responses wear scar diameter and coefficient of friction were analysed with grey relational analysis. Form figure 8, it is clear that for all loading conditions the maximum value of GRG is observed at 0.5 wt% of Al_2O_3 nanoparticles. The highest value of GRG is observed at 250 N load and 0.5 wt% of Al_2O_3 nanoparticles in base lubricant oil presenting optimal condition for improving the tribological characteristic of base lubricant oil.

4. CONCLUSION

In this study modified Al_2O_3 nanoparticles were dispersed in base lubricant oil and tested for improvement in tribological properties of base oil. On completion of this study following major conclusions are drawn:

1. For all values of loading conditions 0.5 wt% addition of Al_2O_3 nanoparticles in the base lubricant oil has a significant effect on wear preventive characteristic through minimizing wear scar diameter and coefficient of friction.

2. Further addition of Al_2O_3 nano particle in base lubricant oil has adverse effect on its tribological properties.
3. The average reduction in wear scar diameter and coefficient of friction is by 20.75 % and 22.67 % respectively with addition of 0.5 wt% of Al_2O_3 nanoparticles in base lubricant oil.
4. Considering the effect of load along with addition of nanoparticles, grey relational analysis presents 250 N along with 0.5 wt% of Al_2O_3 nanoparticles as an optimal combination for minimizing both wear scar diameter and coefficient of friction.
5. The 0.5 wt% of Al_2O_3 nanoparticles is an optimal value of nano additive with all loading conditions improving wear preventive and friction-reducing characteristic of base oil through minimizing wear scar diameter and coefficient of friction of base oil. This is due to the mending effect, ball-bearing effect and tribofilm formation at interface improving the performance of rolling contact. But with an increase in Al_2O_3 nanoparticle concentration, a large number of nanoparticles obstructs to rolling movement increasing wear rate and coefficient of friction.

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